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# Standard Operating Procedure for Accelerated Corrosion Testing at ARL

by Thomas A Considine

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# **Standard Operating Procedure for Accelerated Corrosion Testing at ARL**

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## **1. Introduction**

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This document is intended to define a standardized process for all accelerated corrosion testing performed by the US Army Research Laboratory (ARL) and its collaborative partners. Instructions for sample preparation and the X-scribe are defined in detail. General chamber operations are discussed, including directives for pauses in operation and deviations from operational parameters of the chamber. Test sample evaluations are explained with respect to both corrosion originating from the scribe and blistering in the field. Finally, the daily and periodic reporting requirements are detailed. By clearly specifying the processes above, ARL and its collaborative partners will limit the number of variables when performing round-robin testing.

## **2. Sample Preparation**

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### **2.1 Pre-Scribe**

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The samples/parts shall be thoroughly free of oil, grease, wax, dirt, scale, and other foreign matter and shall show no visible signs of corrosion products prior to scribing. Clean samples with a dry, lint-free cloth. If the hand wipe is not sufficient, additional cleaning shall be in accordance with (IAW) TT-C-490 Method II or III.

### **2.2 Scribe**

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Samples are to be manually scribed using a tungsten carbide scribe as described in ASTM D1654, Section 5.1.2, to make an "X" scribe (Fig. 1). The scribe tool shall be in good order with a sharp, unbent, and undulled tip that provides clean scribe lines. Scribe width is not to exceed 0.5 mm. Automated scribing procedures may be used so long as proper objective quality evidence can be provided to prove that the automated procedure does not produce results that deviate from the traditional manual procedure. Scribes used on ferrous materials should not be used to scribe nonferrous materials so as to prevent contamination of the substrate during the process. The "X" scribe is made by scribing 2 intersecting lines from one corner to the opposite corner across the face of the sample using a straight metal guide rule. The scribe should initiate and terminate no closer than 1/2 inch from the edge of the sample. Scribes that do not penetrate the coating to the substrate are to be rescribed, taking care to stay within the trough cut by the original scribe. Occasionally, scribes may deviate from a straight line due to surface profile of the substrate, various properties of the coating, or slipping of the guide rule. These scribes are acceptable for use in testing when agreed upon by testing authorities.

Samples are to be wiped, blown with air, or brushed lightly with a soft bristle brush to remove spalled coating and metal flakes from the scribe and surface of the sample.



**Fig. 1** Example of a tungsten carbide scribe tool

### **3. Operation**

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#### **3.1 Exposure**

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Sample exposure will be conducted according to the applicable specification for test procedure(s). These specifications may include GMW14872 Cyclic Corrosion Testing (typically Exterior, Exposure C), ASTM B117 Neutral Salt Fog Testing, ASTM G50 Atmospheric Corrosion Testing, and others. A log will be maintained to document all applicable conditions as described in the appropriate specification (i.e., for ASTM B117, maintain a log of wet and dry bulb temperatures, bubble tower temperature, solution pH, solution specific gravity, salt fog deposition rate, etc.).

#### **3.2 Pauses in Exposure**

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Testing may be interrupted due to extreme weather or other unforeseen circumstances. In the event of a prolonged pause in accelerated corrosion exposure, all the test samples shall be rinsed in deionized (DI) water and the affected chambers closed in an ambient condition. All pauses in testing should be noted in the chamber logs.

#### **3.3 Operating Outside Predefined Parameters**

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All abnormal conditions encountered during testing shall be documented and noted in the chamber logs. If any abnormalities arise, testing shall be suspended until corrections are made.

## 4. Evaluation

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### 4.1 Preparation for Rating

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When rating during selected intervals throughout the sample exposure, panels are to be rinsed in DI water and allowed to dry prior to rating. If using a forced air dry, do so at room temperature. Upon completion of exposure, the panels are to be rinsed in DI water, allowed to dry, and scraped as described in paragraph 8.1.1 of ASTM D1654.

### 4.2 Evaluation of Creepage from Scribe

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Discoloration and blistering originating at the scribe shall be the targeted areas of evaluation. For evaluation of creepage, use an optical measuring tool with minimum  $10\times$  magnification. Observe at least 6 points along each scribe (12 points total per sample) in a practical, uniform distribution. The results from these will be averaged to obtain the overall rating of the panel. Ratings are obtained according to Table 1 in ASTM D1654. The maximum and minimum creepage shall be evaluated from the edge of the scribe. The final 3 mm of each leg of the “X” scribe are to be ignored. Evaluations made at the “V” intersection of the “X” scribe shall be made with respect to creepage that clearly originates from one side of the intersection if possible. If the creepage originates from both sides of the “V” of the intersection, then those recorded values will be halved to account for both origin points (Fig. 2).



**Fig. 2** Creep originating from each side of the scribe (left) and of indeterminate origin (right)

### **4.3 Evaluation of Unscribed Areas**

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Evaluation of the unscribed areas of the sample are to be done IAW TT-C-490. All blistering, delamination, or other corrosive effects originating from the edge of the sample are to be ignored out to 0.5 inch. Ratings are to be determined as described in Table I of TT-C-490.

## **5. Reporting**

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### **5.1 Daily Records**

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Daily records are to be kept for all laboratory testing and entered into log books.

#### **5.1.1 Cyclic Chambers**

Many of the daily records are kept by the cyclic corrosion chambers themselves as internally gathered data. The operator is responsible for measuring the daily collection rate, making note of any abnormalities, and correcting those abnormalities or suspending the test.

#### **5.1.2 Salt Fog Chambers**

The operator is responsible for collecting the following data daily: wet and dry bulb temperatures, bubble tower temperature, fog collection rate, specific gravity, and pH of collected fog. Additionally, the operator is responsible for making note of any abnormalities and correcting those abnormalities or suspending the test.

### **5.2 Periodic Records**

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At a minimum, weekly measurements and observations shall be recorded for all laboratory testing. Measurements and observations for outdoor testing, at periods agreed to between the customer and the testing facility, shall be entered into the appropriate logs.

#### **5.2.1 Cyclic Chambers**

##### **5.2.1.2 Mass Loss Coupons**

Standard mass loss coupons, IAW GMW14872, are to be run alongside all testing in cyclic chambers. Prior to exposure, each mass loss coupon shall be cleaned IAW TT-C-490 Method II or III and weighed in grams to 4 decimal places. Every 10–20 cycles, 2 mass loss coupons should be removed. These coupons shall be rinsed in DI water to remove any salts prior to glass bead blasting. After blasting, the

coupons shall be solvent wiped to remove any remaining dirt and allowed to dry before obtaining a final weight. Mass losses shall be recorded and added to the mass loss record.

#### 5.2.1.3 Solution

Each time a new batch of solution is made, the date, initial pH, and conductivity are to be recorded and added to the chamber log. Immediately prior to making a new batch of solution, a final pH and conductivity reading shall be made and added to the chamber log.

### 5.2.2 Salt Fog Chambers

#### 5.2.2.1 Mass Loss Coupons

Mass loss coupons shall be run strictly IAW Appendix 3 of ASTM B117.

#### 5.2.2.2 Solution

Each time a new batch of solution is made, the date and initial pH are to be recorded and added to the chamber log. Immediately prior to making a new batch of solution, a final pH reading shall be made and added to the chamber log.

## 6. Conclusion

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The process parameters outlined above provide ARL and its partners a solid path forward with respect to generating similar data in accelerated corrosion testing. By using the same cleaning procedures, contamination of the substrate is minimized during the scribe process. All partners adhering to guidelines and reporting on any deviations in exposure duration or chamber conditions allows the potential identification of reasons behind incongruent results. The more subjective nature of the ratings processes described in ASTM D1654 is streamlined, allowing all collaborative partners to make ratings in the same way and lessening data bias. Finally, by adhering to the same reporting standards, another potential cause of dissimilar results can be evaluated with greater ease.

## 7. References

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- ASTM B117-16. Standard practice for operating salt spray (fog) apparatus. West Conshohocken (PA): ASTM International; 2016.
- ASTM D1654-08(2016)e1. Standard test method for evaluation of painted or coated specimens subjected to corrosive environments. West Conshohocken (PA): ASTM International; 2016.
- ASTM G50-10(2015). Standard practice for conducting atmospheric corrosion tests on metals. West Conshohocken (PA): ASTM International; 2015.
- GMW14872. Cyclic corrosion laboratory test. General Motors Worldwide: 2013.
- TT-C-490F. Chemical conversion coatings and pretreatments for metallic substrates (base for organic coatings). Federal Specifications and Standards: 2013.

## List of Symbols, Abbreviations, and Acronyms

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ARL	US Army Research Laboratory
ASTM	American Society for Testing and Materials
DI	deionized
GMW	General Motors Worldwide
IAW	in accordance with
pH	potential of hydrogen

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